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Indian Standard

CALCIUM STEARATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY — SPECIFICATION

भारतीय मानक

विस्फोटक और आतिशबाजी सामग्री उद्योग हेतु केल्शियम स्टिएरेट — विशिष्टि

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

November 1989

Price Group 2

Explosives and Pyrotechnics Sectional Committee, CDC 51

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 15 April 1989, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Calcium stearate has a molecular formula $CaC_{36}H_{70}O_4$ and a molecular mass of 606. There is already an Indian Standard IS 2519 : 1983 'Specification for calcium stearate for cosmetic industry', but the requirements for calcium stearate for explosives and pyrotechnic industry are different from that for cosmetic industry, which has led to formulation of this standard. Calcium stearate is mainly used as a lubricating agent in the explosives and pyrotechnic industry.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CALCIUM STEARATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for calcium stearate for explosives and pyrotechnic industry.

2 REFERENCES

IS No.	*Title*
IS 323 : 1959	Specification for rectified spirit (*revised*)
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a fine white bulky powder free from visible extraneous impurities and gritty matter.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the table.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

4.2 Marking

The container shall be marked with the following information:

a) Name of the material;
b) Net mass;
c) Name of the manufacturer and recognized trade mark, if any;
d) Batch or lot number in code or otherwise; and
e) Date of manufacture.

Table 1 Requirements for Calcium Stearate for Explosives and Pyrotechnic Industry

(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test. Ref to Clause No. in Annex A
(1)	(2)	(3)	(4)
i)	Fineness — passing through 63 micron IS Sieve, percent by mass, *Min*	100	A-2
ii)	Moisture, percent by mass, *Max* (at 100 ± 2°C)	4·0	A-3
iii)	Total ash, percent by mass, *Max*	9·5	A-4
iv)	*Purity*		
	a) From stearic acid on dry basis, percent by mass, *Min*	95·0	A-6
	b) From total calcium on dry basis, percent by mass, *Max*	104*	A-5
v)	Free fatty matter, percent by mass, *Max*	1·0	A-7
vi)	Test for acidity or alkalinity:		
	a) Acidity as H_2SO_4, percent by mass, *Max*	0·05	A-8
	b) Alkalinity to phenolphthalein	Nil	A-8

*The purity from total calcium on dry basis exceeds 100 due to the presence of other calcium impurities which are calculated as calcium stearate.

5 SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(*Clause* 3.2)

ANALYSIS OF CALCIUM STEARATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF SIEVE ANALYSIS

A-2.1 Procedure

Place about 50 g of the material, accurately weighed, in 63 micron IS sieve and wash by means of a slow stream of butanol from a wash bottle until all the material that is capable of passing through the sieve has passed. Let the butanol from the sieve drain and then dry the sieve containing the residue on a steam bath. If there is any residue, carefully transfer it on to a tared watch glass and dry to constant mass at 105 ± 2°C.

A-2.2 Calculation

$$\text{Material retained on 63 micron IS sieve, percent by mass} = \frac{100\, M_1}{M_2}$$

where

M_1 = mass in g of the residue retained on the specific sieve,

M_2 = mass in g of the material taken for the test.

A-3 DETERMINATION OF MOISTURE

A-3.1 Procedure

Weigh accurately about 3 g of the material and dry in an oven at 100 ± 2°C for 3 hours. Note the mass of the dried material.

A-3.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100\, M_2}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = loss in mass in g on drying.

A-4 DETERMINATION OF TOTAL ASH

A-4.1 Procedure

Weigh accurately about 10 g of the dried material in a porcelain dish and gently heat until the organic matter is burnt away. Increase the temperature to about 1 100°C until all the carbon residue is burnt away. Allow to cool and weigh. Repeat the operation of heating, cooling and weighing until constant mass is obtained.

A-4.2 Calculation

$$\text{Total ash, percent by mass (on dry basis)} = \frac{100\, M_2}{M_1}$$

where

M_2 = mass in g of the ash, and

M_1 = mass in g of the dry material taken for the test.

A-5 DETERMINATION OF CALCIUM STEARATE — PURITY (FROM TOTAL CALCIUM ON DRY BASIS)

A-5.1 Reagents

A-5.1.1 *Hydrochloric Acid* (HCl), (1:1 *v/v*).

A-5.1.2 *Ammonia*

A-5.1.3 *Ammonia — Ammonium Chloride Buffer*

Add 142 ml of concentrated ammonia solution (sp gr 0·88 - 0·90) to 17·5 g of ammonium chloride. Dilute to 250 ml with distilled water.

A-5.1.4 *Ethylene Diamine Tetra Acetic Acid* (EDTA), M/10 solution (37·225 g/l disodium salt).

A-5.1.5 *Solochrome Black Indicator*, (0·10 g in 100 ml rectified spirit).

A-5.2 Procedure

Dissolve the ash obtained (**A-4.1**) in 30 ml 1 : 1 HCl and make up the volume with distilled water to 500 ml. Take 50 ml of this solution and neutralize with ammonia solution. Add 5 ml of the buffer and 5 drops of indicator and titrate with M/10 EDTA solution.

A-5.3 Calculation

$$\text{Purity from total calcium on dry basis} = \frac{TV \times 60{\cdot}67}{M}$$

where

TV = titre value of M/10 EDTA, and

M = mass of the material taken for ash determination.

A-6 PURITY FROM STEARIC ACID

A-6.1 Reagent

A-6.1.1 *Hydrochloric Acid*, (1:1 *v/v*).

A-6.1.2 *Dichloromethane*

A-6.2 Procedure

Weigh accurately about 2·0 g of the sample and transfer to a glass beaker. Add 25 ml of 1:1 HCl. Digest the solution for one hour on a hot plate. Cool it to room temperature and transfer into a separating funnel. Extract with dichloromethane (25 ml). Repeat the extraction thrice and wash the dichloromethane extract with water till free from acid. Take the dichloromethane extract in a tared evaporating dish and evaporate the solvent on a water bath. Repeat the operation of heating, cooling and weighing until constant mass (*M*) is obtained.

A-6.3 Calculation

$$\text{Purity as calcium stearate (on dry basis)} = \frac{53{\cdot}35 \times M \times 100}{100 - (\text{percent moisture})}$$

where

M = mass in g of residue.

A-7 DETERMINATION OF FREE FATTY MATTER

A-7.1 Reagent

A-7.1.1 *Dichloromethane*

A-7.2 Procedure

Weigh accurately about 5 g of the material and mix it with 100 ml of dichloromethane in a beaker. Shake for about half an hour and filter. Take the filtrate in tared porcelain dish and evaporate slowly on a steam bath. Cool and weigh the residue.

A-7.3 Calculation

$$\text{Free fatty matter, percent by mass} = \frac{100\, M_2}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the residue.

A-8 TEST FOR ACIDITY OR ALKALINITY

A-8.1 Reagents

A-8.1.1 *Rectified Spirit*, conforming to IS 323 : 1959.

A-8.1.2 *Phenolphthalein Indicator Solution*

Dissolve 0·10 g in 100 ml of 60 percent rectified spirit.

A-8.1.3 *Methyl Orange*

Dissolve 0·01 g in 100 ml of water.

A-8.1.4 *Standard Hydrochloric Acid*, 0·10 N.

A-8.2 Procedure

Suspend 10 g of the material in 50 ml of rectified spirit and shake for half an hour. Filter the solution and make up the volume to 100 ml with rectified spirit. Take 25 ml of this solution and add 5 drops of phenolphthalein indicator, if alkalinity (red coloration) is observed, titrate with N/10 hydrochloric acid. If no alkalinity (red coloration) is observed, add 5 drops of methyl orange indicator to another aliquot of the solution. If orange red coloration is observed, titrate against N/10 sodium hydroxide.

A-8.3 Calculations

$$\text{Percent alkalinity as sodium carbonate} = \frac{TV \times 0{\cdot}005\,3 \times 100 \times 4}{10} = TV \times 0{\cdot}212\,0$$

$$\text{Percent acidity as sulphuric acid} = \frac{TV \times 0{\cdot}004\,9 \times 100 \times 4}{10} = TV \times 0{\cdot}196\,0$$

where

TV = titre value.

ANNEX B

(*Clause* 5.1)

SAMPLING OF CALCIUM STEARATE FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight, glass or other suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling including the date of sampling and the year of manufacture of the material.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot separately for ascertaining the conformity of the material to the requirements of this specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be as given in Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clauses* B-2.2 *and* B-3.1.1)

Lot Size N (1)	Number of Containers to be Selected n (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them 1, 2, 3,……, r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot till the requisite sample is obtained.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument, a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **3** and shall not exceed 1 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 1 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three test samples representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as the referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and set of individual samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

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